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(54) PROCESS FOR THE PREPARATION OF VEGETABLE POWDER INSTANTLY SOLUBLE OR MICRODISPERSIBLE IN WATER

(71) We, INSTITUT DE RECHERCHES APPLIQUEES AUX BOISSONS I.R.A.B. a French Body Corporate of 87, Rue de Paris, 93 Montreuil, France and BUREAU ETUDES RECHERCHES EXPLOITATION DE BREVETS B.E.R.E.B. a French Body Corporate of Rue du Pave, 78-Epone, France, do hereby declare the invention for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

The present invention relates to the production of dehydrated vegetable material instantly soluble or micro-dispersible in water.

It is known that dry materials comprising a vegetable extract, especially a fruit juice, and sugar can be prepared industrially, the vegetable extract being first of all dehydrated, for example by lyophilisation, and then mixed intimately with the other solid ingredients e.g. sugar. The dry materials so obtained have the disadvantage that they are not homogeneous and their aroma content is often low.

Moreover, it is known that dehydration to a residual humidity content of not less than 3% can be achieved by various processes other than lyophilisation (e.g. vacuum spray drying or atomisation in a current of hot air). These processes have the disadvantage that they last too long and/or alter the aroma of the material being dried.

The use of ultra-high frequency heating to dehydrate *in vacuo* material which has been made into a foam has the advantage that drying time is reduced and the aroma is better preserved. In fact, this method ensures that there is a homogeneous temperature distribution in the material to be dried, and the latter is not contacted with a heating surface or subjected to the action of a radiant heating panel (thus avoiding localised areas of overheating). Furthermore, it is less costly than lyophilisation, which in any case cannot be used industrially if the initial content of solids exceeds 40%, because of the difficulties of freezing the material before sublimation, and of cooling the condensers during sublimation.

U.S. Patent No. 2,555,450 describes an apparatus and process for dehydrating animal or vegetable materials. It discloses the possibility of incorporating a thickener such as sugar if the material to be dehydrated is not sufficiently viscous, and recommends a method of heating which is not an ultra-high frequency method. The formation of foams would be an obstacle to the satisfactory running of this process.

Similarly the dielectric methods of heating disclosed in U.S. Patents Nos. 2,436,966 and 3,432,636 have the same disadvantage, which is inherent in the nature of the microwaves employed.

French Patent No. 1,354,111 discloses rapid drying of perishable substances by means of a micro-wave field applied *in vacuo* to organic matter either in the frozen state or in solution or suspension in a liquid near the freezing point. Such a method, which especially suffers from the technological difficulties involved (i.e. simultaneous action of cold, a vacuum, and micro-waves), cannot be applied industrially.

The present invention provides a process which mitigates or avoids these difficulties, and gives a dried product having a substantially unaltered smell or taste.

The present invention provides a process for the dehydration of a composition comprising a vegetable material which comprises subjecting the composition in liquid or pulp form and having a solids content of at least 50% by weight to an ultra-high frequency field having a frequency of at least 850 MHz at a pressure of 1 to 20 mm. Hg., the pressure and frequency of the field

being such that the field strength is less than the critical discharge voltage at that pressure the viscosity and/or gas content of the composition being such that the compositions foams during subjection to the said pressure and loses water, and wherein there is a homogeneous temperature distribution through the composition during dehydration and a dehydrated product is formed which is instantly soluble or microdispersible in water.

In the new process, the wet vegetable material to be dried is sufficiently viscous and/or aerated to form a foam under the vacuum. The ultra-high frequency field having a frequency of at least 850 MHz and preferably of 2450 MHz, in the interior of the resonant cavity in which the material is dehydrated always has a field strength less than the critical discharge voltage under the pressure in the cavity.

The material to be dried should desirably have a sufficient initial viscosity so that foaming takes place when the composition is heated by the high frequency field. The natural thickness present in the vegetable material will often impart a sufficient viscosity to the composition but if this is not sufficient, viscosity can be adjusted by adding a thickening agent. Suitable thickeners for adding to the composition are carbohydrates glucides, sucroglycerides, carragheens, gum arabic, pectintates, methylcellulose, carboxymethylcellulose, and proteins, especially albumin. Of the carbohydrates, sucrose has proved valuable for two reasons: firstly, it enables vegetable extracts to be more easily expanded or formed into a foam under a vacuum; secondly, it improves the solubility of the dry product.

The desired viscosity can also be obtained more easily by initially concentrating the substance to be treated, for example by freezing or by inverse osmosis. Also, a calculated proportion of dry product from a previous operation may be added to the material to be dried. It is of course possible to combine the various means for increasing the viscosity with one another, without departing from the scope of the invention.

A current of nitrogen or carbon dioxide can also be passed into the viscous mixture to provide an occluded gas which can subsequently expand to form the foam.

The content of solids in the mixture to be dehydrated is preferably between 50 and 85% by weight.

The viscous mixture, which has optionally been treaed as above with nitrogen or carbon dioxide, is distributed in layers of low or medium thickness (1 to 20 mm) on a plate of a chamber under vacuum and exposed to the ultra-high frequency field of at least 850 Mhz, preferably equal to 2450 MHz. The plate which is in a vacuum oven can be either fixed or movable, but in either case the heating period is between a few minutes and a few tens of Minutes. Depending on the thickness of the layer formed on the one handfi and the pressure value on the other hand, the heating period may last, in an apparatus which is working in cycles, for between 4 and 30 minutes.

During the heating phase an expansion of the composition, which is a pasty mass, is observed, the apparent volume being increased by the formation of a foam which soon solidifies as it loses the larger part of its initial content of water. No change in colour in the mass obtained is observed at this stage, nor any appearance of a crust.

In the succeeding phase the ultra-high frequency field is preferably removed while the vacuum is maintained in order to continue the desiccation, and at the same time the average temperature of the mass is lowered as a result of this action. This period can last, depending on the circumstances, for a few seconds to a few minutes. The heating can also be intermittent.

At this stage, the desiccation as such as complete. The vacuum is released, preferably by introducing a current of inert gas such as nitrogen, and then the spongy mass, which still has a certain plasticity on account of its temperature, is cooled by a suitable means until it has a friable consistency.

This mass of dehydrated product is then broken up, and then packaged, in a dry atmosphere, in closed containers which are hermetically sealed against water vapour and air.

The heating under a vacuum by means of the ultra-high frequency field may be carried out in two phases. The temperature of the mass to be dehydrated is below 30°C during the first phase of the desiccation (removal of 75% of the initial quantity of water) at the end of which the residual moisture content is at most equal to 20% by weight of the total mass. The temperature of the material remains below 50°C during the second phase of the desiccation, at the end of which the residual moisture content is not more than 3% by weight.

During the whole dehydration process in the ultra-high frequency field, care must be taken to see that the electrical field strength within the dehydration chamber does not exceed a maximum value, called the discharge voltage, which would give rise to an intense ionisation of the gases in the said chamber. The value of this critical voltage depends on the frequency and on the pressure. In practice, at 915 MHz it is necessary to operate above 15 mm Hg if it is desired to have a sufficiently strong field without any risk of initiating an arc. However,

at 2450 MHz it is sufficient to maintain the pressure slightly above 2 mm Hg. The high frequencies are thus more useful since they enable stronger fields to be employed without requiring very low pressures to be used, and thus more energy to be introduced into the resonant cavity.

In general, any vegetable material can be dried by the new process, but the invention is especially useful for drying vegetable extracts such as infusions, decoctions, juices, pulp and essential oils, particularly fruit juices and pulped fruit. The new process is in fact the only one which has enabled powder of small fruits such as blackcurrants, raspberries and strawberries to be obtained, which are instantly soluble in water, and preserve their aroma. The new process is also the only one which has enabled a powder of aniseed essence to be obtained which is instantly microdispersible in water.

The products of the new process, which are themselves novel, in general have a residual moisture content of less than or equal to 3% by weight, and contain a) from 1 to 80% by weight (expressed as dry weight) of vegetable substance, and b) from 16 to 99% by weight of a thickener, such as a carbohydrate, glucide, sucroglyceride, carragheen, gum arabic, egg albumin or a mixture thereof.

A suitable ultra-high frequency oven for use in the new process is that designed and manufactured by Messrs. L.M.I. (Les Micro-ondes Industries). It compriseh a stabilised high voltage supply unit (4600 volts); supplying a unit for creating micro-waves, consisting of a 2.5 kW magnetron having an anode cooled by water circulation, the magnetron being connected to an antenna situated 15 cm above the microwave window in the oven; the oven comprising a horizontally arranged cylindrical chamber of stainless steel, provided with two inlets, two borosilicate glass inspection windows, and in particular, in its upper part, with a window made of polypropylene, which is transparent to micro-waves. Metal partitions within this chamber define the resonant cavity, the geometry of which is designed to avoid the formation of electric arcs.

This apparatus is also provided with an arrangement for generating a vacuum, and with mechanism for regulating the vacuum to between 1 and 20 mm Hg.

The Examples which follow illustrate the invention.

EXAMPLE 1

Dehydration of a coffee extract

20 g of a coffee extract containing 50% by weight of solids are placed in an evacuated chamber and subjected to the action of an ultra-high frequency field of 2450 MHz for 6 minutes, under a pressure of 5 mm Hg; the product is then allowed to stand for 5 minutes under a vacuum of about 1 mm Hg. A porous solid is obtained, having an apparent volume which is double the initial volume, which, after grinding, gives a powder which is easily soluble in hot water.

EXAMPLE 2

Preparation of a blackcurrant powder

After removing the stalks, berries of fresh or thawed blackcurrent are finely ground to form a pulp and then mixed with an equal weight of sucrose. The solids content is then between 55 and 60% by weight. 1% of albumin by weight of the mixture is added.

30 grams of this paste are spread out over a surface area of 180 cm², and then placed in a vacuum. A large stable expansion (to 10 times the initial volume) is observed. An intermittent ultra-high frequency field of 2450 MHz is then applied, the vacuum remaining below 15 mm Hg (the total heating period is 9 minutes in a time interval of 20 minutes). A product having the appearance of a friable meringue is thus obtained, which, after grinding, gives a light powder which is instantly dispersible in water.

EXAMPLE 3

Preparation of a strawberry powder

Fresh or thawed strawberries are finely ground in the presence of an equal weight of sucrose and 2.25% of citric acid to produce a paste of solids content between 55 and 60% by weight. A thin film of this paste is laid on the plate of the micro-wave oven (20 g/180 cm²). An ultra-high frequency field of 2450 MHz is then applied for 4 minutes, the loss of bubbles of water vapour causing an expansion to 3 to 4 times the initial volume. After 5 minutes a dry product is obtained, which, after grinding, provides a power, instantly dispersible in water, which allows a beverage having the colour and taste of the fresh fruit to be prepared.

EXAMPLE 4

Preparation of a raspberry powder

Fresh or thawed raspberries are finely ground in the presence of twice their weight of sucrose and 3% of citric acid by weight relative to the mixture of ground raspberries and sucrose to produce a paste whose solids content is between 75 and 85% by weight. A thin film of this paste (40 g/180 cm²) is laid on the plate of the micro-wave oven. The vacuum is 15 mm Hg at the start of the operation and 1 mm Hg at the end of the cycle. The film is dried for 5 minutes by means of an ultra-high frequency field

produced by a 500 W source. The initial expansion observed subsides but the rapid loss of bubbles of water vapour leads to the expansion to 2 to 3 times the initial volume. After grinding, the product yields a powder instantly dispersible in water which, provides a beverage which retains a good aroma of raspberries.

The loss of vitamin C during the course of the drying operation has been determined. Before drying, the paste contained 20 mg of ascorbic acid per 100 g solids and drying reduces this value to 17.5 mg, that is to say a loss of vitamin C of 12.5% by weight relative to the weight of vitamin C before drying.

Examination of the absorption spectrum in visible light of an aqueous solution of this powder shows no qualitative or quantitative change compared with the starting mixture: the maximum remains at 515 mμ with the same optical density.

EXAMPLE 5

Whole orange powder

A mixture of 17 g of a paste of raw peel from which the bitterness has been removed by enzyme treatment, 60 ml of orange juice, 110 g of sucrose, 6 g of citric acid, and 0.6 g of carragheens, having a solids content of 67 to 72% by weight, is homogenised in a grinder.

A thin layer of the paste obtained is laid on the plate of the micro-wave oven. The layer is then dried under a vacuum of less than 15 mm Hg by means of an ultra-high frequency field of 2450 MHz, for 5 minutes. Towards the end of the operation a second expansion (2-3 times the initial volume), due to the rapid loss of water vapour is observed. In 10 minutes a dry and porous product is obtained, which redissolves easily in water after it has been ground, and preserves an aroma of oranges which is unchanged by the treatment.

EXAMPLE 6

On carrying out the process as indicated in Example 5, using other citrus fruits (juice and paste of the peel), the same results as regards dissolving were obtained with lemon, grape-fruit, mandarin orange and clementine powders.

EXAMPLE 7

Orange juice powder

Orange juice is first concentrated to 80-85% by weight of solids, and to 100 g of concentrate, 70 g of sucrose and 4 g of citric acid are added. After homogenisation, a thin layer of this paste is placed on the plate of the micro-wave oven. The paste is dehydrated under a vacuum of 15 mm Hg at the start of the cycle, using an ultra-high frequency field produced by a 100 W source and then a 300 W source. After 9 minutes, a dry product has formed which can be ground to provide a powder. The loss of ascorbic acid during the course of this operation is only 4% by weight relative to the weight of vitamin C before drying.

EXAMPLE 8

Powder of orange juice

1 g of carragheen and ground orange pulp (150 g) are added to 100 g of orange juice concentrate (to give a mixture of solids content 50-55% by weight). 20 g of this mixture are dehydrated under a vacuum below 10 mm Hg by means of an ultra-high frequency field of 2450 MHz applied intermittently. A dry and porous product is obtained after 15 minutes, which is reduced to powder.

EXAMPLE 9

Powder of aniseed extract (fennel, star anise, green anise).

100 g of sugar are dissolved in 50 ml of an aqueous dispersion of 1.5 g of aniseed essence. 0.2 g of sucroglyceride is added. The mixture is homogenised and 20 g of the resulting paste are then spread out on the plate of the micro-wave oven (180 cm^2) and subjected to a vacuum. An expansion is observed, which subsides. The product is then subjected to an ultra-high frequency field of 2450 MHz for 7 minutes, while maintaining the vacuum at a value below 10 mm Hg. In the last minutes of the cycle a large expansion (five times the initial volume) is observed. 13.5 g of a dry product are thus obtained, which, after grinding, provides a powder which is micro-dispersible in water and is pleasantly perfumed. On adding water to the aniseed powder, an instant and homogeneous suspension is obtained, the opalescence of which is identical to that which is observed when water is added to an aniseed liqueur.

By proceeding as indicated in Example 9, powders which are flavoured with mint, verbena, camomile, balm mint and linden, have been obtained.

EXAMPLE 10

Granadilla powder

50 parts by weight of sugar and 0.1 part by weight of egg albumin are added to 50 parts by weight of granadilla puree. The mixture is made perfectly homogeneous and the paste is cooled to $-10°C$. 100 g of this paste are introduced into the resonant cavity, and spread over a surface of 750 cm^2, and a vacuum is generated down to 4 mm Hg. A homogeneous foam 2-3 cm. high is thus formed, and microwaves are admitted for 30 minutes, the anode current at the magnetron being 0.2A. A dry product is thus obtained which is easily soluble, and which retains all the aroma of the fruit.

WHAT WE CLAIM IS:—

1. A process for the dehydration of a composition comprising a vegetable material which comprises subjecting the composition in liquid or pulp form and having a solids content of at least 50% by weight to an ultra-high frequency field having a frequency of at least 850 MHz at a pressure of 1 to 20 mm. Hg., the pressure and frequency of the field being such that the field strength is less than the critical discharge voltage at that pressure, the viscosity and/or gas content of the composition being such that the composition foams during subjection to the said pressure and loses water, and wherein there is a homogeneous temperature distribution through the composition during dehydration and a dehydrated product is formed which is instantly soluble or microdispersible in water.

2. Process according to claim 1 wherein the composition is one to which a thickener has been added.

3. Process according to claim 2, in which the thickener is a carbohydrate, glucide, sucroglyceride, carragheen, gum arabic, egg albumin or a mixture thereof.

4. Process according to claim 3 wherein the pressure is 1 t 15 mm. Hg.

5. Process according to any one of the preceding claims, in which the vegetable material is an infusion, a decoction, a juice, a pulp or an essential vegetable oil.

6. Process according to claim 5 in which the vegetable material is strawberry, raspberry, blackcurrent or granadilla juice or pulp, orange, lemon, grapefruit, mandarin orange or clementine juice or pulp, coffee or aniseed ssence.

7. Process according to any one of the preceding claims in which the said ultra-high frequency field is a field of frequency 2450 MHz.

8. Process according to claim 1 substantially as described in Example 1.

9. Process according to claim 1 substantially as described in any one of Examples 2-9.

10. Process according to claim 1 substantially as hereinbefore described.

11. A dehydrated powder produced by the process of any of the preceding claims.

12. A dehyrated powder produced by the process of any one of claims 1-3 and 8.

13. A dehydrated powder produced by the process of claim 4 or 9.

14. A dehydrated powder according to any one of claims 11-13 having a residual moisture content of less than or equal to 3% by weight and containing a) 1 to 80% by weight (expressed as dry weight) of vegetable material and b) 16 to 99% by weight of thickener.

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